



## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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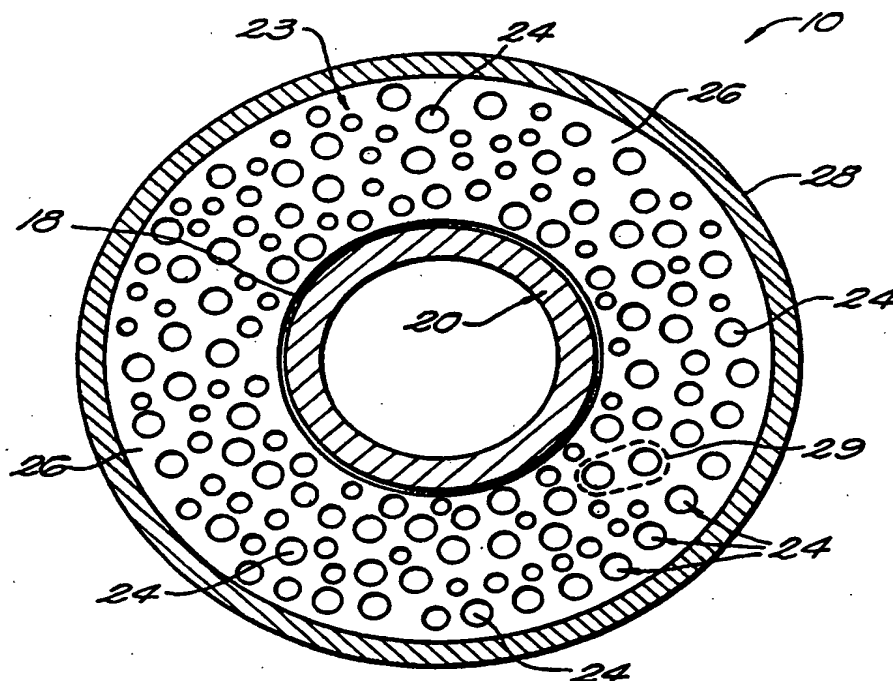
## Published

*With international search report.**Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.*

(54) Title: SUBSEA PIPELINE INSULATION

## (57) Abstract

A deep sea insulated pipeline comprises an inner pipe (20) which is encased lengthwise by an insulating core (23). The insulating core comprises macrospheres (24) surrounded by syntactic foam that includes a semi-rigid resin binder and microspheres (27). The semi-rigid resin binder reinforces the macrospheres (24) to provide sufficient strength to withstand the hydrostatic pressure at depths in excess of several thousand feet of water, and is yet flexible enough to accommodate bending associated with deep sea pipe laying operations. The deep sea insulated pipeline may also include a protective outer casing. The inner pipe (20) extends through and cooperates with the outer casing to define an annulus chamber containing the insulating core (23). The outer casing may be a plastic pipe. In a preferred embodiment the semi-rigid resin binder includes Bisphenol-A epoxy resin, an anhydride curing agent, and a flexibilizer.



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## SUBSEA PIPELINE INSULATION

### BACKGROUND OF THE INVENTION

The invention relates to the field of insulated pipelines, and in particular to the  
5 field of subsea pipelines suitable for use in deep water.

The resistance to flow of liquid products such as oil increases as temperature decreases. This problem can be reduced by using thermally insulated pipelines. However, for offshore pipelines it has usually been more cost effective to reduce the need for insulation by injecting various chemicals into the product.

10 However, more and more oil and gas is being recovered in deeper, colder water, from subsea production systems where use of viscosity reducing chemicals requires a dedicated line to transport them to the wellhead. This, combined with the fact that the cost of insulating pipelines typically increases with depth, indicates that insulated pipelines are most expensive where the alternatives are least attractive.

15 Prior art insulation used in undersea pipelines include porous plastic foam, such as polyurethane foam. As known, the lower the density of this insulating material, the higher percentage of air within the material, and therefore the more efficient it is as an insulator. However, as the insulating ability of the material increases due to decreased density, the weaker the material becomes. Specifically, as the density decreases so does  
20 the depth at which the foam cellular structure can operate in. Generally, prior art insulators fail in a few hundred feet of water due to the hydrostatic pressure on the insulation. So the design tradeoff comes down to how light an insulator can be placed onto the surface of the pipe and have it withstand the hydrostatic pressure and other stresses, and at the same time provide the necessary thermal insulation for a long period  
25 of time.

These prior art insulators worked in the past because the operational depth of the pipeline was rather shallow. However, the oil industry has undergone a vary rapid movement into deeper water. Several years ago the deepest producing oil well was in approximately fifteen hundred feet of water. The deepest oil well producing today is in  
30 four thousand feet of water. The deepest producing oil well planned for two years from today is in ten thousand feet of water. Significantly, as the operating depth increases these relatively lightweight, low cost, low strength prior art materials become unsuitable.

Specifically, the materials can no longer withstand the hydrostatic pressure and become saturated with water, thus undesirably becoming a thermal conductor rather than an insulator.

The use of syntactic foams has been discussed as an insulator suitable for deep-sea pipeline insulation. As known, syntactic foams are composite materials in which hollow structures, such as microspheres are dispersed in a resin matrix. However, in any practical manufacturing situation microspheres can not be introduced into the foam in a sufficient quantity to provide the requisite thermal insulation. In addition, the resin binders which hold the microspheres in conventional syntactic foams are too rigid to sustain the bending associated with conventional pipeline laying techniques.

Therefore, there is a need for an insulator which provides sufficient insulation for deep sea operation, and yet is flexible enough to withstand the bending associated with pipe laying operations.

## 15 SUMMARY OF THE INVENTION

An object of the present invention is to provide an insulator suitable for deep sea operation.

Briefly, according to the present invention, a deep sea insulated pipeline comprises an inner pipe which is encased lengthwise by an insulating core. The insulating core comprises macrospheres surrounded by syntactic foam that includes a semi-rigid resin binder and microspheres. The semi-rigid resin binder reinforces the macrospheres to provide sufficient strength to withstand the hydrostatic pressure at depths in excess of several thousand feet of water, and is yet flexible enough to accommodate bending associated with deep sea pipe laying operations.

25 The deep sea insulated pipeline may also include a protective outer casing. The inner pipe extends through and cooperates with the outer casing to define an annulus chamber containing the insulating core. The outer casing may be a plastic pipe.

In a preferred embodiment the semi-rigid resin binder includes Bisphenol-A epoxy resin, an anhydride curing agent, and a flexibilizer. In the syntactic foam these ingredients may have parts-by-weight (pbw) of approximately 100 pbw, 100 pbw, and 35-45 pbw, respectively, for 35 pbw of microspheres, where the ratios are with respect to the weight of the syntactic foam.

The macrospheres are preferably fiberglass, sealed, seamless, hollow, air and water tight spheres of about 0.25 - 0.5 inches in diameter. Other high strength advanced composite type fibers (e.g., carbon fibers, aramid, etc.) may also be used rather than fiberglass.

5        Advantageously, the deep sea pipeline insulation of the present invention has insulating properties 2-10 times better than the prior art as measured according to ASTM standard C-518, and yet is sufficiently flexible to sustain the bending which is known to occur in conventional pipe laying. That is, the syntactic foam is a partially flexible material that combines sufficient rigidity to support the macrospheres with conformal  
10    ability approaching that of an elastomeric insulator.

These and other objects, features and advantages of the present invention will become apparent in light of the following detailed description of preferred embodiments thereof, as illustrated in the accompanying drawings.

## 15    **BRIEF DESCRIPTION OF THE DRAWINGS**

FIG. 1 illustrates a pictorial illustration of a deep sea pipe laying operation;

FIG. 2 illustrates a cross sectional view of an insulated pipe according to the present invention;

FIG. 2A illustrates an exploded cross sectional view of the insulating core and  
20    macrospheres within the annulus;

FIG. 3A-3B together provide a flow chart illustration of a method for insulating a length of pipe;

FIG. 4 is a lengthwise cross sectional view of the insulated pipeline;

FIG. 5 is a cross sectional view taken along lines 5-5 in FIG. 4; and

25    FIG. 6 is a cross sectional view of an alternative embodiment insulated pipe.

## **DETAILED DESCRIPTION OF THE INVENTION**

FIG. 1 is a pictorial illustration of deep sea pipeline laying operation. Lengths of insulated pipe 10 are interconnected on a lay barge 12 and the lengths exit the barge  
30    along a stinger 14 which is a downwardly curved pipe exit ramp. As the barge 12 moves along, the insulated pipeline eventually comes to rest on the ocean floor. Significantly, as the pipeline is being laid it is subject to a first bend 16 near the surface of the water

and a second bend 18 above the ocean floor. This technique is often referred to as an "S-curve lay".

FIG. 2 is a cross sectional illustration of the insulated pipe 10. The insulated pipe 10 includes an inner pipe 20 that is typically steel and has a diameter of about 4 to 6 inches and a wall thickness typically 0.25 to 0.5 inches. The pipe 20 is often referred to as "flow line" because oil or gas, or in most cases a combination of the two pass through the pipe. To protect the pipe 20 from the corrosive effects of sea water, a thin anti-corrosion barrier of plastic film 18 or a paint like coating covers the exterior of the pipe 20. Outside of that is an insulating core 23 that comprises a plurality of fiber reinforced macrospheres 24 surrounded by a syntactic foam 26.

FIG. 2A illustrates a cross sectional view of an exploded area 29 shown in FIG. 2. The area 29 includes macrospheres 24 and the syntactic foam 26. The syntactic foam 26 includes a semi-rigid resin binder 25 containing microspheres 27. It should be noted that when viewed in three dimensions, the macrospheres actually contact adjacent macrospheres, and the voids between these spheres are filled by the syntactic foam 26.

The syntactic foam 26 reinforces the macrospheres 24 to provide sufficient strength to withstand the hydrostatic pressure at depths in excess of several thousand feet of water, and is yet flexible enough to accommodate the bending associated with deep sea pipe laying operations. The insulating core 23 has a radial thickness ranging from 1 to 3 inches. The thickness is dependent upon the depth at which the pipeline is designed to operate. The semi-rigid syntactic foam shall be discussed in detail hereinafter.

The macrospheres 24 are generally spherical shaped and have a diameter of about 0.25 to 0.5 inches. The walls are preferably fiberglass and have thickness dependent upon the operational depth. Specifically, the greater the operational depth of the pipeline, the greater the wall thickness required to sustain the hydrostatic pressure at that depth. For example, at depths where the hydrostatic pressure is a thousand psi or less, the wall thickness may be quite thin (e.g., 0.01 inches). In contrast, at ten thousand feet where the hydrostatic pressure approaches 5,000 psi the wall thickness is increased significantly (e.g., 0.03 inches). It is contemplated that other high strength advanced composite type fibers (e.g., carbon fibers, aramid, etc.) may also be used rather than fiberglass.

The microspheres 27 interspersed within the semi-rigid resin binder 25 are typically about 100 microns in diameter (i.e., 0.004") hollow spheres generally containing a gas which may be atmospheric air, although it may be richer in nitrogen than atmospheric air. The microspheres 27 have a wall thickness of about one micron.

5 As known, the microspheres are manufactured by blowing glass in a furnace in the presence of blowing agents that cause the glass to bubble.

To provide the desired strength to withstand the crushing hydrostatic pressure, the macrospheres 24 are interspersed throughout the syntactic foam 26. Significantly, the syntactic foam 26 surrounds the macrospheres to provide a semi-rigid resin matrix  
10 which raises the strength of the macrospheres in contrast to their strength in the absence of the syntactic foam. For example, testing has shown that the macrospheres 24 exhibit a crush strength approximately two times greater when encapsulated in syntactic foam, then they would otherwise exhibit if unreinforced. The insulated pipeline 10 also includes a protective outer casing 28 (e.g., a polypropylene pipe) which is approximately 12" in  
15 diameter. We shall now discuss a process for insulating a deep sea pipe.

FIGs. 3A and 3B together provide a flow chart illustration of a method for insulating a deep sea pipe. In step 40 the exterior surface of a length of pipe (e.g., forty feet) is cleaned to remove corrosion and scale products. The cleaning may be performed by sandblasting the metallic pipe to provide a clean, bare steel substrate to work with.

20 Step 42 is then performed to apply an epoxy anti-corrosion coating to the surface of the cleaned pipe. As a result, the exterior is a clean plastic surface which is well bonded to the steel pipe.

Referring now to FIGs. 3A, 4 and 5, a plurality of centralizer supports 45 are then attached at several places along the cleaned length of pipe 20. The centralizers 45  
25 are spider-like devices that coaxially center the inner pipe 20 within the outer pipe 28.

In step 46 the outer pipe 28 is drawn over the centralizer supports 45 to thereby define an annulus 47 between the inner pipe 20 and the outer pipe 28. The outer pipe 28 is preferably several feet shorter than the inner pipe 20 in order to leave 12-24 inches of the inner pipe exposed on each end. The radial depth of the annulus may be about 1 to  
30 3 inches, which represents the radial distance between the exterior of the inner pipe and the interior of the outer pipe.

Step 48 is then performed to apply caps at both ends of the covered pipe to seal

the annulus. The capped pipe is then inclined 30-45° in step 50. In step 52 the fiberglass macrospheres 24 (FIG. 2) are deposited through an opening in the upper cap until the macrospheres fill the annulus 47. The pipe may be agitated slightly with a vibrator. Alternatively, the operator may occasionally strike the pipe with a soft headed hammer to provide the necessary agitation to facilitate the flow of macrospheres through the annulus. In step 54 the upper cap is then sealed and a vacuum pump is attached to remove the air from the annulus through a filtered opening in the upper cap.

Referring now to FIG. 3B, in step 56 a mixer/reservoir containing the semi-rigid syntactic foam is attached to an opening in the lower cap through a conduit. The foam is then injected under pressure from the mixer into the bottom of the annulus. The vacuum pump attached to the filtered opening in the upper cap draws the syntactic foam through the annulus, filling the voids between the macrospheres. When the foam starts to exit through the filtered opening in the top, the annulus is full. The pipe is then lowered to the horizontal position, the opening in the caps are sealed, and the pipe is placed into an oven where it is baked at about 200°F for 4-8 hours, preferably about 6 hours, to cure epoxy within the resin binder. When the epoxy is cured, the end caps are removed and the resultant structure is a length of insulated pipe as shown in cross section in FIG. 2.

The semi-rigid syntactic foam includes several preferred ingredients which are identified in Table 1, along with the relative amount of each ingredient.

**TABLE 1**

INGREDIENTS	TOTAL RANGE (parts-by-weight)
Bisphenol-A Epoxy Resin	100 pbw
Anhydride Curing Agent	85 - 150 pbw
Glass Microspheres	25 - 50 pbw
Flexibilizer (e.g., Polybutadiene or Polyglycol)	25 - 55 pbw

In a preferred syntactic foam embodiment, the Bisphenol-A epoxy resin is 100 pbw, the anhydride curing agent is 100 pbw, the glass microspheres are 35 pbw and the



flexibilizer is 35-45 pbw.

Significantly, the syntactic foam is strong enough to support the macrospheres and provide the requisite crush strength, while still flexible enough to sustain the bending while being laid. It is contemplated that various flexibilizers will provide the syntactic  
5 foam with the conformal ability approaching that of an elastomeric insulator. The "*Handbook of Epoxy Resin*" written by H. Lee and K. Neville and published by McGraw-Hill Book Company discusses a number of different flexibilizers.

To form the pipeline, lengths of insulated pipe are welded together. As shown in FIG. 4 a short distance (12- 24 inches) of the inner pipe 20 is left uncovered by the  
10 insulating core 23 (FIG. 2) and the outer pipe 28. Molded half collars (not shown) of the insulating core material are then placed around the welded ends, and polyethylene or polypropylene is flame sprayed over the secured half collars. As a result, continuous lengths of insulated pipe can be provided. Generally, four lengths of pipe are welded together to make what is called a quadjoint, covered with insulating half collars and then  
15 flame sprayed with the polyethylene or polypropylene to provide a 160 foot insulated pipeline that is barged to the laying barge and connected to another 160 foot length piece on the laying barge.

FIG. 6 is a cross sectional view of an alternative embodiment insulated pipe 50.

This embodiment is substantially the same as the embodiment illustrated in FIG. 2, with  
20 the exception of the protective outer casing 28 (FIG. 2). Specifically, it is contemplated that the pipeline may be laid in a shallow man-made trench on the ocean floor. As a result the protective outer casing may not be required.

To manufacture the alternative embodiment insulated pipe 50, a mold may be placed around the inner pipe analogous to the protective pipe. The method set forth in  
25 FIGs. 3A and 3B can then be used to insulate the inner pipe, and following the curing step the mold would be remove leaving the cured insulating core at the exterior of the pipe.

One of ordinary skill will recognize that the present invention is clearly not limited to the pipe sizes discussed herein or the materials. For example, it is clearly  
30 contemplated that a wide variety of pipes of a wide variety of sizes and thickness will find it advantageous to use the insulator disclosed herein. In addition, it is contemplated that the syntactic foam may be extruded onto the inner pipe.

Although the present invention has been shown and described with respect to several preferred embodiments thereof, various changes, omissions and additions to the form and detail thereof, may be made therein, without departing from the spirit and scope of the invention.

5

What is claimed is:

## CLAIMS

- 1 1. An insulated pipeline, comprising:  
2 an inner pipe; and  
3 an insulating core that encases lengthwise said inner pipe and comprises  
4 macrospheres surrounded by syntactic foam that includes a semi-rigid resin binder and  
5 microspheres, wherein said semi-rigid resin binder reinforces said macrospheres to  
6 provide sufficient strength to withstand the hydrostatic pressure at depths in excess of  
7 several thousand feet of water, and is yet sufficiently flexible to accommodate bending  
8 associated with deep sea pipe laying operations.
- 1 2. The insulated pipeline of claim 1, wherein said semi-rigid resin binder comprises  
2 Bisphenol-A epoxy, an anhydride curing agent and a flexibilizer.
- 1 3. The insulated pipeline of claim 1, wherein said syntactic foam includes about 35  
2 parts-by-weight (pbw) of said microspheres, and about 100 pbw of Bisphenol-A epoxy  
3 resin, 85-150 pbw of anhydride curing agent, 25-55 pbw of flexibilizer.
- 1 4. The insulated pipeline of claim 3, further comprising a plurality of centralizers  
2 which space said inner pipe and said outer pipe to define said annulus.
- 1 5. The insulated pipeline of claim 3, wherein said insulating core has a radial  
2 thickness greater than one inch.
- 1 6. The insulating pipe of claim 5, wherein said outer pipe is several feet shorter than  
2 said inner pipe to leave a portion of said inner pipe exposed on each end.
- 1 7. The insulating pipe of claim 1, further comprising a cylindrical outer protective  
2 casing through which said inner pipe extends coaxially, establishing an annulus between  
3 said inner pipe and said outer protective casing, wherein said insulating core is located  
4 within said annulus.
- 1 8. A method of insulating a pipe for deep sea operation, comprising the steps of:

2        inserting an inner pipe through a larger diameter outer casing, and positioning the  
3        inner pipe coaxially within the larger diameter outer casing to form an annulus radially  
4        between the inner pipe and the outer pipe;  
5        providing an insulating core that fills the annulus, by  
6        adding a quantity of macrospheres into the annulus to fill the annulus;  
7        inserting a semi-rigid curable syntactic foam into the annulus such that the  
8        foam encases and reinforces the macrospheres to provide sufficient strength to  
9        withstand the hydrostatic pressure at depths in excess of several thousand feet of  
10       water, and yet accommodate the bending associated with deep sea pipe laying  
11       operations; and  
12       curing the syntactic foam.

1       9.       The method of claim 8, further comprising the step of preparing the semi-rigid  
2       syntactic foam by mixing about 35 parts-by-weight (pbw) of microspheres, about 100  
3       pbw of Bisphenol-A epoxy resin, approximately 85-150 pbw of anhydride curing agent,  
4       and about 25-55 pbw of flexibilizer.

1       10.      The method of claim of claim 9, further comprising the steps of cleaning the inner  
2       pipe and depositing an anti-corrosion barrier on the exterior of the inner pipe, prior to  
3       said step of inserting the inner pipe through the larger diameter outer casing.

1       11.      The method of claim 9, further comprising the step of placing at least one  
2       centralizer on the inner pipe to coaxially position the larger diameter outer casing relative  
3       to the inner pipe in order to form the annulus.

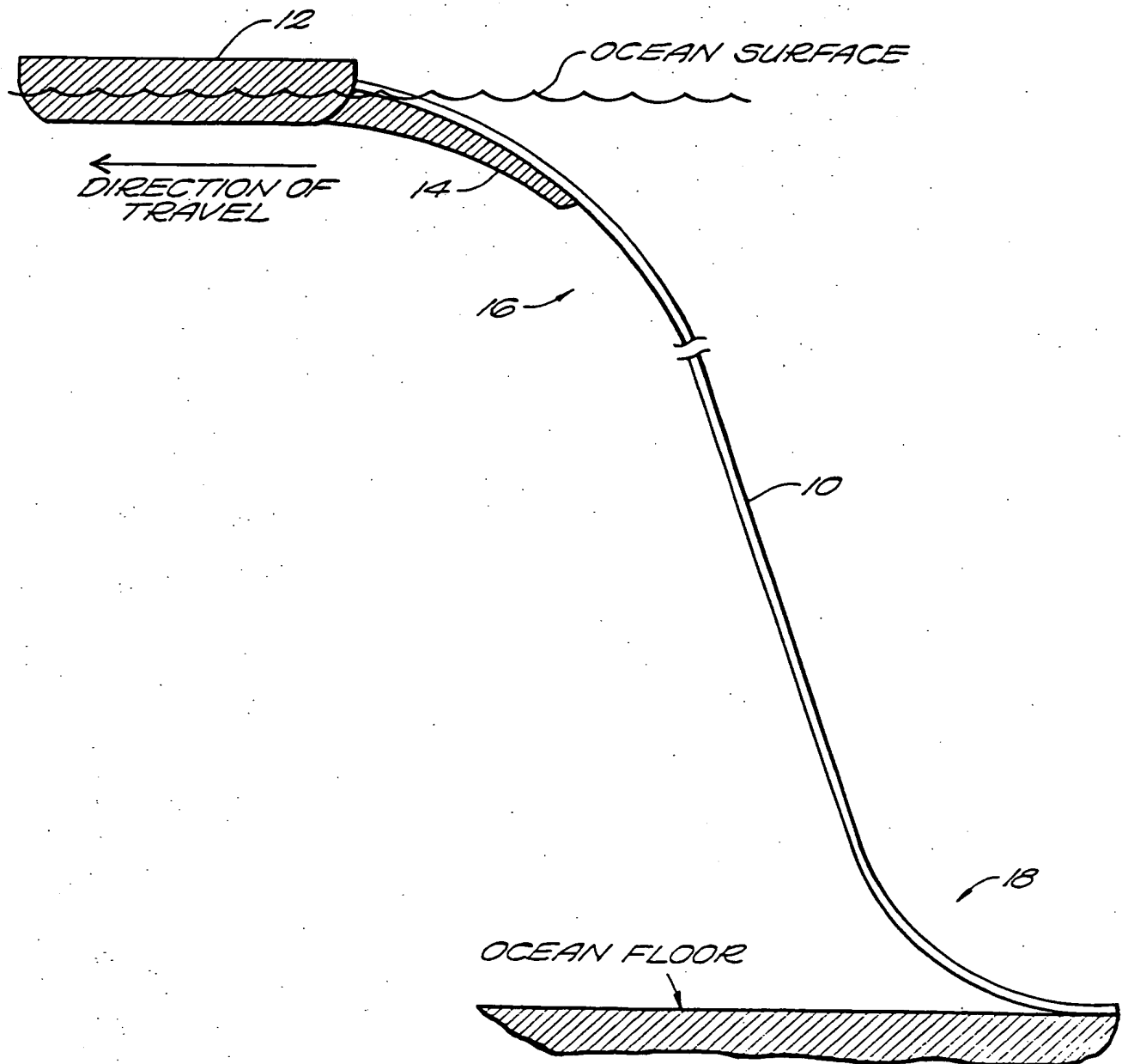
1       12.      The method of claim 11, wherein said large diameter outer casing is a pipe.

1       13.      The method of claim 11, further comprising the step of removing the larger  
2       diameter outer casing following the step of curing.

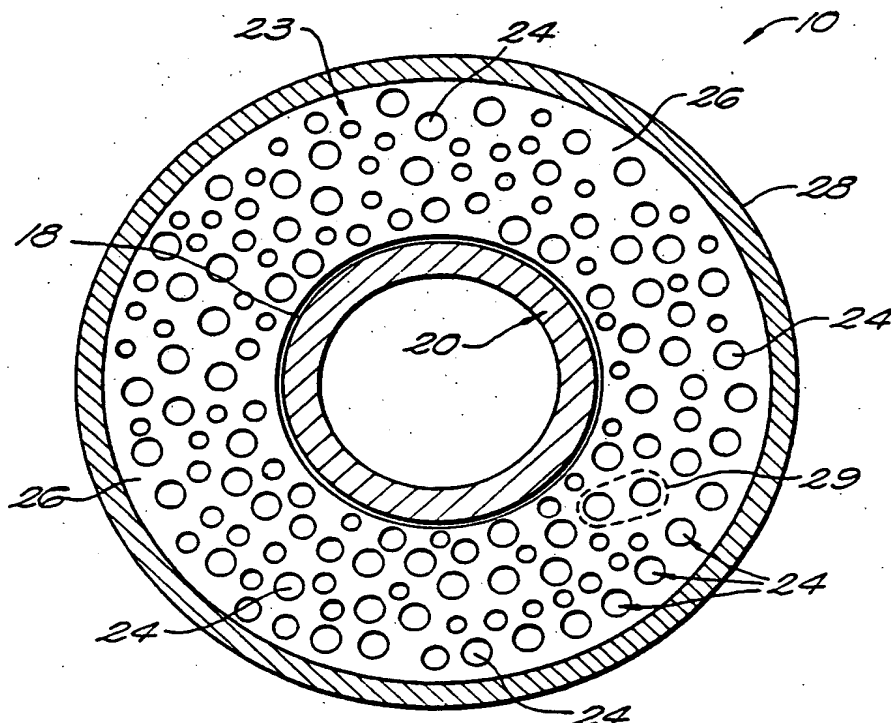
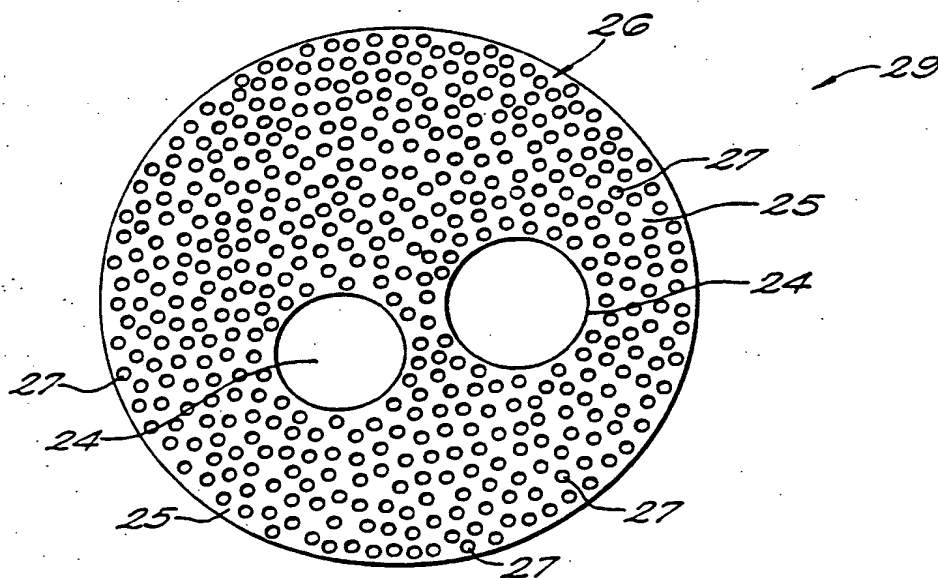
- 11 -

- 1 14. A semi-rigid syntactic foam for use in deep sea operations, comprising about 100
- 2 parts-by-weight (pbw) of microspheres, about 100 pbw of Bisphenol-A epoxy resin,
- 3 approximately 85-150 pbw of an anhydride curing agent and about 25-55 pbw of a
- 4 flexibilizer.

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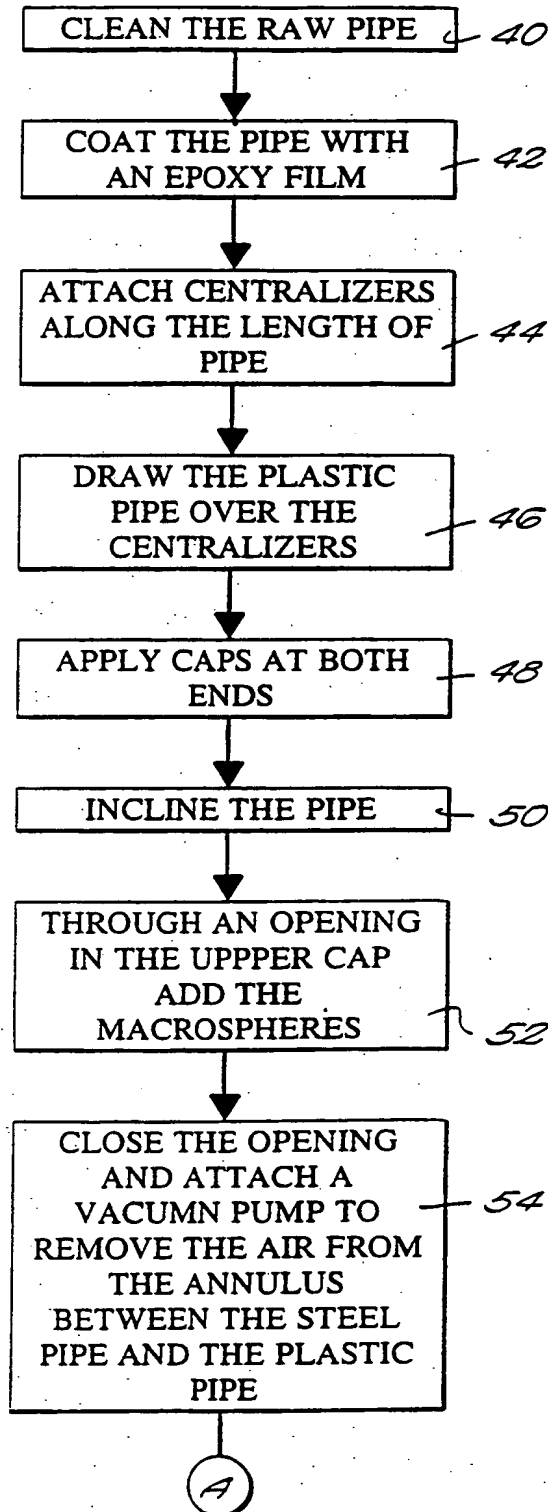
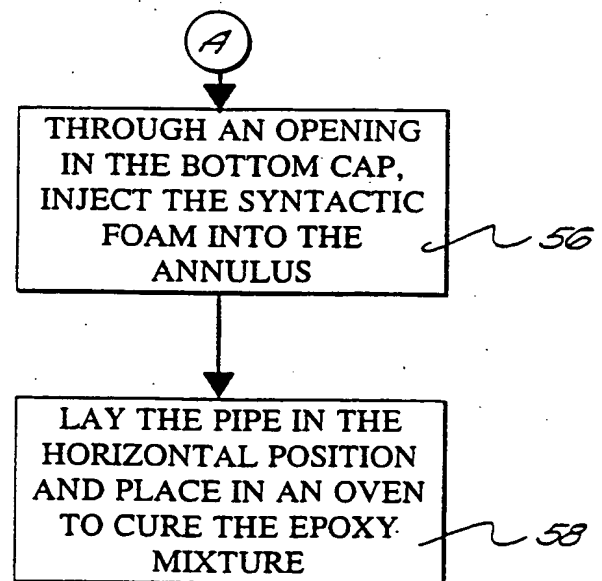
**FIG. 1**

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**FIG. 2****FIG. 2A**

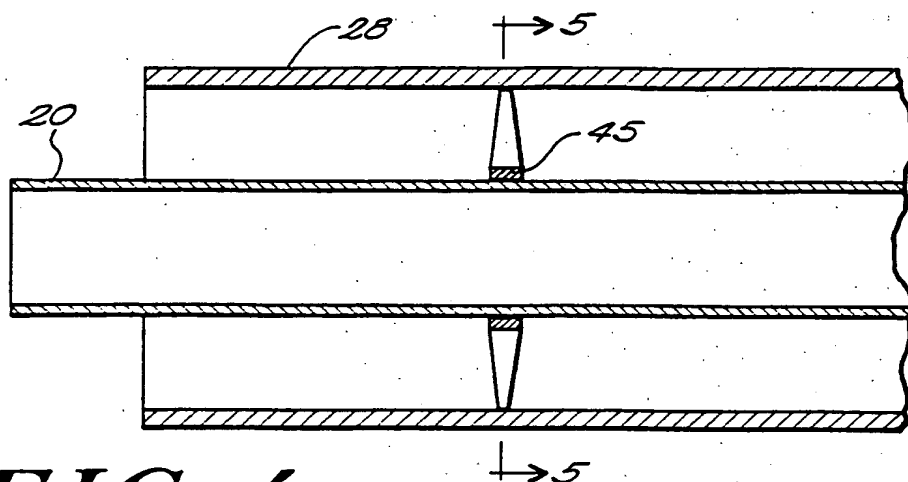
SUBSTITUTE SHEET (RULE 26)

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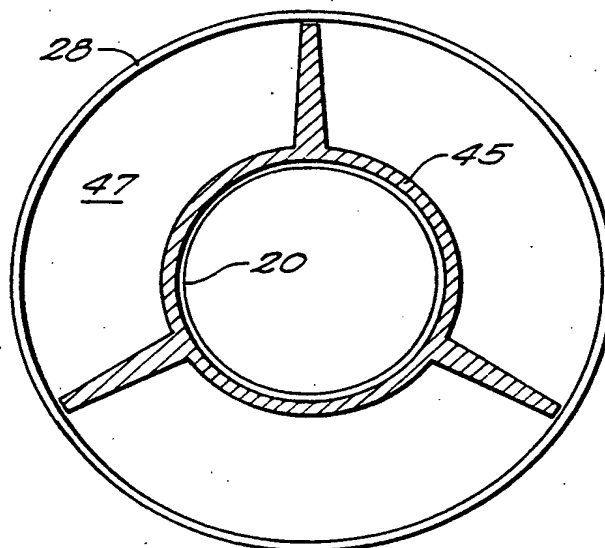
*FIG. 3A**FIG. 3B*



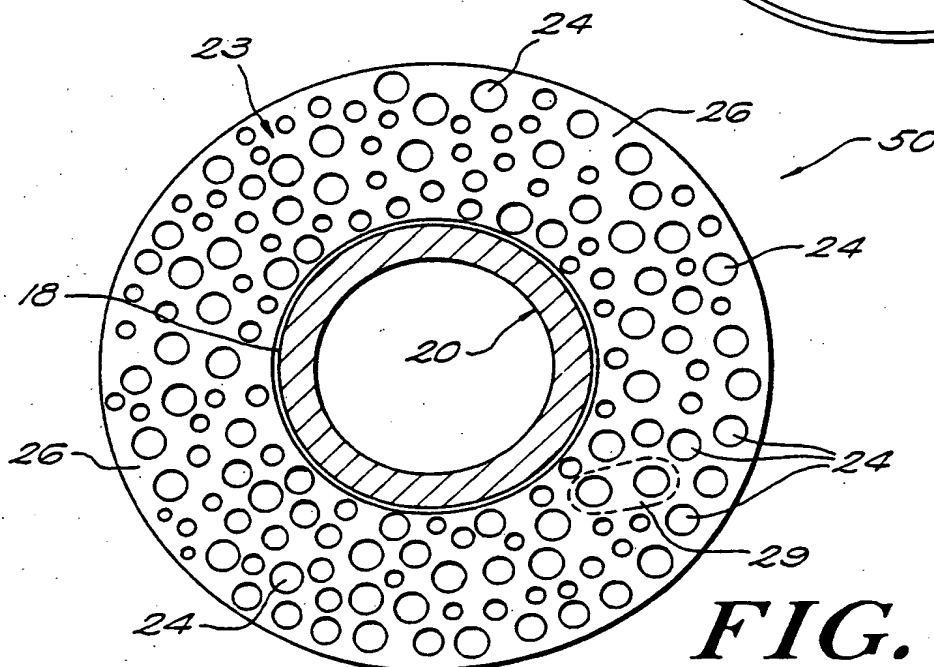
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**FIG. 4**



**FIG. 5**



**FIG. 6**

# INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 98/15201

**A. CLASSIFICATION OF SUBJECT MATTER**

IPC 6 F16L59/02 B29C67/20 C08J9/32

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 F16L B29C C08J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4 348 243 A (CRAUBNER INGO) 7 September 1982 see the whole document ---	1-13
X	US 3 996 654 A (JOHNSON ARNE I) 14 December 1976 see the whole document ---	1-6
A	US 3 622 437 A (HOBICA EDWARD C ET AL) 23 November 1971 see the whole document ---	1-13
A	US 4 482 590 A (BOULEY ROBERT W ET AL) 13 November 1984 see column 3, line 17 - column 3, line 54; figures 1,2 --- -/--	1-6,8,9, 11

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

**\* Special categories of cited documents:**

"A" document defining the general state of the art which is not considered to be of particular relevance

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"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

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Date of the actual completion of the international search

10 November 1998

Date of mailing of the international search report

23/11/1998

Name and mailing address of the ISA

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## INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 98/15201

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	GB 2 244 490 A (INST FRANCAIS DU PETROL) 4 December 1991 see claims 7-9; example 3 -----	1-3,8,9
X	US 4 568 603 A (OLDHAM SUSAN L) 4 February 1986	14
A	see column 3, line 64 - column 5, line 11 -----	2,3,9
A	US 4 595 623 A (DU PONT PRESTON S ET AL) 17 June 1986 -----	2,3,9,14

# INTERNATIONAL SEARCH REPORT

International application No.

PCT/US 98/15201

## Box I Observations where certain claims were found unsearchable (Continuation of item 1 of first sheet)

This International Search Report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:  
because they relate to subject matter not required to be searched by this Authority, namely:
2. ☐ Claims Nos.:  
because they relate to parts of the International Application that do not comply with the prescribed requirements to such an extent that no meaningful International Search can be carried out, specifically:
3. ☐ Claims Nos.:  
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

## Box II Observations where unity of invention is lacking (Continuation of item 2 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

see separate sheet

1. ☐ As all required additional search fees were timely paid by the applicant, this International Search Report covers all searchable claims.
2. ☒ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this International Search Report covers only those claims for which fees were paid, specifically claims Nos.:
4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this International Search Report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

### Remark on Protest

- ☐ The additional search fees were accompanied by the applicant's protest.
- ☐ No protest accompanied the payment of additional search fees.

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/US 98/15201

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 4348243 A	07-09-1982	DE 2747579 A FR 2406778 A GB 2009181 A, B	26-04-1979 18-05-1979 13-06-1979
US 3996654 A	14-12-1976	NONE	
US 3622437 A	23-11-1971	DE 2022493 A FR 2042523 A GB 1278895 A	12-11-1970 12-02-1971 21-06-1972
US 4482590 A	13-11-1984	NONE	
GB 2244490 A	04-12-1991	FR 2662634 A CA 2043724 A JP 4232034 A	06-12-1991 01-12-1991 20-08-1992
US 4568603 A	04-02-1986	AU 565356 B AU 4238785 A CA 1239750 C DE 3563631 A EP 0181368 A WO 8505113 A	10-09-1987 28-11-1985 26-07-1988 11-08-1988 21-05-1986 21-11-1985
US 4595623 A	17-06-1986	AU 565356 B AU 4238785 A CA 1239749 A DE 3563631 A EP 0181368 A JP 61501211 T WO 8505113 A	10-09-1987 28-11-1985 26-07-1988 11-08-1988 21-05-1986 19-06-1986 21-11-1985

